Syllabus MSNE 411+511 Fall 2020 rev. 25Aug2020 PJL

Metallography and Phase Relations – The main areas of focus are sample preparation, visible light microscopy, scanning electron microscopy, and x-ray microanalysis. We will also study the microstructures which occur in various metals plus a few ceramics and composites, as well as non-equilibrium processing which produce those microstructures, and how failures result from improper microstructure. Required for the BS-MSNE.

- **Instructor:** Peter Loos **office:** ME 215 **Office hours or Zoom meeting:** by appointment
- **Teaching Assistant:** to be determined, probably none
- **Class Meets:** TuTh 1:30-2:50 in KCK 101. The tentative plan is for lab exercises, if possible, from about 12:45 to 1:30 during about six weeks of the semester.
- **Prerequisites:** MSNE 301 is required. Completion of MSNE 303 is also assumed.
- **Textbook:** None. There will be several reading assignments from the published literature including a few from ***ASM Handbook*** http://ezproxy.rice.edu/login?url=https://dl.asminternational.org/books/pages/Handbooks_by_Volume especially the volumes ***Metallography and Microstructures***, ***Alloy Phase Diagrams***, ***Materials Characterization***, ***Fractography***. and ***Failure Analysis and Prevention***.
- **Grading:** The plan is 40% homework, 25% tests, 15% quizzes, and 20% presentations. There will be approximately two tests, two quizzes, nine homework assignments, and one presentation. If for some reason part or all of the class must go online, this plan will have to change a bit. 511 students will be assigned a few more problems than 411 students. Points may be deducted for assignments turned in late and for bad or missing references. Some of the quiz questions will be about vocabulary words specific to the practice of metallography and various types of phase diagrams and transformation diagrams.
- **References:** When using information from a source other than class lecture slides or your own original work, a proper reference must be given. Use a format similar to ACS pubs.acs.org/doi/pdf/10.1021/bk-2006-STYG.ch014 or in simpler form: http://library.williams.edu/citing/styles/acs.php.
- **Honor Code:** **For every assignment** students must do all the writing, typing, calculating, and analyzing themselves, must **not** look at or copy from any work done by any student in this class (past or present), and must include and sign the Rice Honor Pledge on work turned in. Plagiarism and other academic fraud will be addressed per the Honor System Handbook. **For homework and presentations,** students are encouraged to communicate with each other, the Instructor, or anyone else with knowledge of the subject. But **for tests and quizzes** no communication (of words, text, numbers, or images) is allowed except with Instructor.
- **Absence Policy:** Students are expected to attend class and lab. Missed quizzes, presentations, and labs must be made up right away. The slides posted on Canvas do not include everything said in class.
- **Software:** Students will use spreadsheets for some of the calculations on homework and tests as well as slide presentation software for the oral presentations. Microsoft Office, LibreOffice, and Apache OpenOffice are generally suitable. Google software is generally not. For compatibility, all files should be saved in either XLS, DOC, PPT, or PDF format.
- **Disability Support:** Any student with a disability requiring accommodations in this class needs to contact both the instructor and the Disability Resource Center in Allen Center.

Lecture Topics

- History of microscopy, metallurgy, and metallography including H.C. Sorby's sample preparation and microscopy in 1863 and H.W.B. Rozeboom's Fe-Fe_3C phase diagram in 1900.
- Alloys of interest: superalloys, carbon steels, stainless steels, aluminum, titanium, WC-Co metal matrix composite, etc.
- Length scales of interest from nanometers to meters.
- Purposes of metallography: research, process and product development, quality and reliability assurance, yield improvement, process control, etc.
- Sample preparation techniques: sectioning, mounting, grinding, polishing, and etching (as needed) to reveal the true structure without changing it. Etchants for specific applications
- Safety in the metallurgy lab: machinery issues as well as corrosive and poisonous chemicals
- Macrostructure / macroetch / macroanalysis of cast and wrought materials. Dye penetrant and magnetic particle inspection, sulfur print and related methods, hydrogen evolution, blue etch anodizing of Ti alloys.
- Visible light microscopy: optical components, magnification, resolution, enhancing resolution through colored filters and oil immersion, bright field/dark field, polarizing filters, interference contrast.
- Scanning electron microscopes: electron guns; thermionic and field emission; electron wavelength; resolution, magnetic lenses; condenser system; raster scan; optimizing spot size. Beam alignment and astigmatism.
- Beam-specimen interaction, scattering of electrons by atoms – six scattering mechanisms, electron trajectories, ranges for electron penetration and x-ray generation.
- Generation & properties of: backscattered, secondary, & Auger electrons; characteristic & Bremsstrahlung x-rays;

cathodoluminescence, voltage contrast, magnetic contrast, thermal wave (scanning electron acoustic) microscopy
- X-ray microanalysis: quantitative analysis, ZAF corrections, minimum detectable amount of an element, energy dispersive vs. wavelength dispersive x-ray detectors, optimizing characteristic x-ray production
- SEM sample preparation for various purposes, surface replicas.
- Kikuchi lines and electron backscatter diffraction (EBSD) (probably have to skip this one)
- Quantitative metallography: ASTM E112 grain size, lognormal grain size distribution, defects, etc.
- Hot work, cold work, and recrystallization. Electron channeling contrast.
- Binary phase diagrams: eutectic, eutectoid, peritectic, peritectoid, monotectic, etc.; cooling paths, precipitates formed. Miscibility gap, congruent melting. Coring. Dendritic structures. Pearlite and lamellar structures.
- Isothermal transformation and continuous cooling transformation diagrams for various alloys, and their use.
- The five strengthening mechanisms.
- Carburizing, nitriding, and decarburization. (probably have to skip this one)
- The hierarchy of authoritative documents. These include plenty of optical and other forms of metallography but not SEM, TEM, EDX, or WDX.
- Field failures: sample preservation, types of failures, and how to recognize them. (might have to skip this one)
- Identifying engineering problems.

Lab exercises: to be determined based on student interest, availability of equipment & specimens, etc.

(1) Sputtering of Pd onto non-conducting SEM specimens such as rocks, egg shells, other ceramics, bugs, and leaves using the Emitech K550 coater on GRB 2nd floor. Principles of sputtering, materials issues, safety, and equipment operation. See video.

(2) Basic SEM operation, observation of the sputtered specimens, using the FEI Quanta 400 in Butcher 160 and its Everhart Thornley secondary electron detector. Comparison of coated and uncoated samples of the same material to demonstrate specimen charging.

(3) Voltage contrast microscopy of an operational amplifier circuit as it switches slowly between two states. Purposes of voltage contrast microscopy. Observation of multiple levels of metal interconnect on the chip as their voltage varies with time using the FEI Quanta 400. Issues with beam voltage, beam current and grounding of the specimen.

(4) Calibration of EDAX Element silicon "drift" type detector energy and energy resolution using Al and Cu samples in the FEI Quanta 400 in Butcher 160. Check for artifacts in the spectrum: escape and sum peaks.

(5) Backscatter electron imaging in the SEM and the three principal contrast mechanisms: topography, atomic number, and channeling.

Other possible lab exercises, to be determined: (a) Sample cleaning and/or surface replication. (b) EDX point analysis, line scans, and area maps. Use of ZAF correction with EDX data. (c) Electron backscatter diffraction for polycrystalline specimens. (d) Wavelength dispersive x-ray analysis using crystal spectrometers.

Exercises (2)-(5), & (b) use the FEI Quanta 400 and EDAX in Butcher 160A. (c) uses FEI Helios and EBSD in Brockman B05. (d) uses JEOL JXA-8530F in KWG 329-330.

Goals for this course:

To address the required ABET Student Outcomes, students will have an ability to:

1. identify, formulate, and solve complex engineering problems by applying principles of engineering, science, and mathematics
3. an ability to communicate effectively with a range of audiences
6. an ability to develop and conduct appropriate experimentation, analyze and interpret data, and use engineering judgment to draw conclusions

and to address one of the ABET Program Criteria, this course prepares graduates to:

- integrate the understanding of the scientific and engineering principles underlying the four major elements of the field: structure, properties, processing, and performance ...

More specifically, students will become familiar with

a) sample preparation methods and equipment for microstructure and macrostructure evaluation
b) microscopy methods and equipment used to observe those microstructures, particularly the visible light microscope (VLM) and scanning electron microscope (SEM) .
c) many of the microstructures which occur in metals and alloys, how those result from composition and thermal history of the material,
d) use of equilibrium phase diagrams and nonequilibrium transformation diagrams to produce and understand microstructure and thermal history
e) the terminology used to describe microstructures
f) how problems in the microstructure or macrostructure can lead to failure

References

ASM Handbook online, see links above
Metallographic Etching *by* G. Petzow, ASM (1978)
Metallography of Phase Transformations by G. A. Chadwick, Butterworths (1972) which is out of print
Annual Book of ASTM Standards standards for metallography. See search results. Fondren Library has all the 2016 volumes on the shelf.
ASTM Special Technical Publications (STP) about metallography. See search results. Fondren has many of these on the shelves.
SAE documents concerning metallography, see approximately 355 search results of various kinds, including numerous Aerospace Materials Standards (AMS).
Scanning Electron Microscopy and X-Ray Microanalysis: A Text for Biologists, Materials Scientists, and Geologists 2nd Ed. by Goldstein, Newbury, Echlin, Joy, Fiori, & Eric Lifshin (1981)
Electron Microscopy and Analysis 3rd Ed. by Goodhew, Humphreys, & Beanland (2001)

Resources On the Web

- Binary equilibrium phase diagrams, which don't need much translated from Russian: www.himikatus.ru/art/phase-diagr1/diagrams.php Or see instructor's hard copy.
- Interactive binary phase diagrams with accompanying photomicrographs from Virginia Tech and Cambridge Univ.: www.sv.vt.edu/classes/MSE2094_NoteBook/96ClassProj/examples/kimcon.html (use the Wayback Machine) www.doitpoms.ac.uk/miclib/phase_diagrams.php
- Microscopy U from Nikon www.microscopyu.com

Journals:

www.sciencedirect.com.ezproxy.rice.edu/science/journal/00260800	Metallography
www.sciencedirect.com.ezproxy.rice.edu/science/journal/10445803	Materials Characterization
link.springer.com.ezproxy.rice.edu/journal/volumesAndIssues/11508	The Physics of Metals and Metallography

Videos:

www.youtube.com/user/MaterialsScience2000	Several good videos listed
www.youtube.com/watch?v=fc8zrgYJCJw	Metallography Part I - Macroscopic Techniques
www.youtube.com/watch?v=UuHofNW40Yw	Metallography Part II - Microscopic Techniques
www.youtube.com/watch?v=GY9lfO-tVfE	SEM
www.youtube.com/watch?v=7Z90OZ7C2jI	Vickers Hardness Test
www.youtube.com/watch?v=xEK-c1pkTUI	Dye Penetrant Inspection

Metallography:

www.msm.cam.ac.uk/phase-trans/abstracts/CP1b.html	Sample preparation for metallography.
www.doitpoms.ac.uk/tlplib/microstructural_exam/	Microstructural Examination
www.doitpoms.ac.uk/tlplib/optical-microscopy/index.php	Optical Microscopy and Specimen Preparation
ASTM E7-03	Standard Terminology Relating to Metallography
ASTM Committee E04 on Metallography	Committee E04 on Metallography
www.astm.org/DIGITAL_LIBRARY/STP/PAGES/STP25092S.htm	History of Committee E4 on Metallography

Metallurgy:

www.doitpoms.ac.uk/tlplib/casting/index.php	Casting
www.doitpoms.ac.uk/tlplib/crystallographic_texture/index.php	Crystallographic Texture
www.doitpoms.ac.uk/tlplib/artefact/index.php	Examination of a Manufactured Article
www.doitpoms.ac.uk/tlplib/phase-diagrams/index.php	Phase diagrams and solidification
www.doitpoms.ac.uk/tlplib/solid-solutions/index.php	Solid Solutions
www.doitpoms.ac.uk/tlplib/solidification_alloys/index.php	Solidification of Alloys